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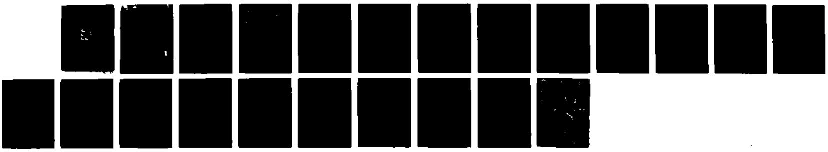

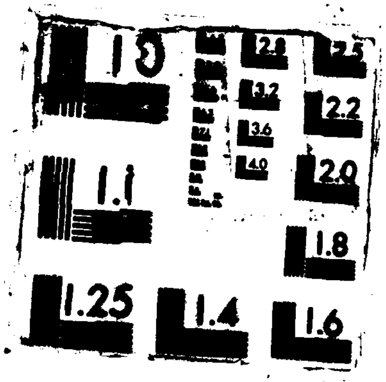
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The Effect of OMCVD Reactor Renovations on GaAs Epilayer Growth

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20 July 1987

Prepared for

SPACE DIVISION
AIR FORCE SYSTEMS COMMAND
Los Angeles Air Force Station
P.O. Box 92960, Worldway Postal Center
Los Angeles, CA 90009-2960

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This report was submitted by The Aerospace Corporation, El Segundo, CA 90245, under Contract No. F04701-85-C-0086-P00016 with the Space Division, P.O. Box 92960, Worldway Postal Center, Los Angeles, CA 90009-2960. It was reviewed and approved for The Aerospace Corporation by M. J. Daugherty, Director, Electronics Research Laboratory.

Capt Richard J. Young/CWX was the project officer for the Mission-Oriented Investigation and Experimentation (MOIE) Program.

This report has been reviewed by the Public Affairs Office (PAS) and is releasable to the National Technical Information Service (NTIS). At NTIS, it will be available to the general public, including foreign nationals.

This technical report has been reviewed and is approved for publication. Publication of this report does not constitute Air Force approval of the report's findings or conclusions. It is published only for the exchange and stimulation of ideas.

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UNCLASSIFIED

SECURITY CLASSIFICATION OF THIS PAGE

REPORT DOCUMENTATION PAGE

1a. REPORT SECURITY CLASSIFICATION
Unclassified

1b. RESTRICTIVE MARKINGS
A183020

2a. SECURITY CLASSIFICATION AUTHORITY

2b. DECLASSIFICATION / DOWNGRADING SCHEDULE

3. DISTRIBUTION / AVAILABILITY OF REPORT
Approved for public release; distribution unlimited.

4. PERFORMING ORGANIZATION REPORT NUMBER(S)
TR-0086A(2925-01)-2

5. MONITORING ORGANIZATION REPORT NUMBER(S)
SD-TR-87-30

6a. NAME OF PERFORMING ORGANIZATION
The Aerospace Corporation
Laboratory Operations

6b. OFFICE SYMBOL *(If applicable)*
AFSD

7a. NAME OF MONITORING ORGANIZATION
Space Division

6c. ADDRESS *(City, State, and ZIP Code)*
El Segundo, CA 90245

7b. ADDRESS *(City, State, and ZIP Code)*
Los Angeles Air Force Station
Los Angeles, CA 90009-2960

8a. NAME OF FUNDING / SPONSORING ORGANIZATION
Space Division

8b. OFFICE SYMBOL *(If applicable)*
AFSTC/WCO

9. PROCUREMENT INSTRUMENT IDENTIFICATION NUMBER
F04701-85-C-0086

8c. ADDRESS *(City, State, and ZIP Code)*
Los Angeles, CA 90009-2960

10. SOURCE OF FUNDING NUMBERS

PROGRAM ELEMENT NO.	PROJECT NO.	TASK NO.	WORK UNIT ACCESSION NO.

11. TITLE *(Include Security Classification)*
The Effect of OMCVD Reactor Renovations on GaAs Epilayer Growth

12. PERSONAL AUTHOR(S)
Donna M. Speckman and Jerry P. Wendt

13a. TYPE OF REPORT

13b. TIME COVERED FROM ______ TO ______

14. DATE OF REPORT *(Year, Month, Day)*
1987 July 20

15. PAGE COUNT
17

16. SUPPLEMENTARY NOTATION

17. COSATI CODES

FIELD	GROUP	SUB-GROUP

18. SUBJECT TERMS *(Continue on reverse if necessary and identify by block number)*
KEY WORDS: gallium arsenide (GaAs) epitaxy
OMCVD

19. ABSTRACT *(Continue on reverse if necessary and identify by block number)*

A major redesign and rebuilding of The Aerospace Corporation's Organometallic Chemical Vapor Deposition (OMCVD) reactor has been carried out. The entire gas-handling system was redesigned and replaced in order to reduce gas contamination, and new high-precision mass flow controllers and temperature baths were installed. Carrier gas flow through the organometallic bubblers was modified for improved safety and reliability, and gas lines that allow for the utilization of several different organometallic gallium and arsenic sources were also installed. Gallium arsenide (GaAs) epilayers grown using trimethylgallium (Me_3Ga) and arsine (AsH_3) in this reactor system following its renovation have consistently exhibited purity levels that are significantly higher than those of epilayers previously grown at The Aerospace Corporation. Epitaxial GaAs films (~4 μm thick) with excellent surface morphologies have been obtained which exhibit carrier concentrations of $3 \times 10^{14}/cm^3$ and 77 K mobilities of approximately 68,000 cm^2/V-s. These physical and electrical characteristics are representative of state-of-the-art unintentionally doped GaAs.

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20. DISTRIBUTION / AVAILABILITY OF ABSTRACT
☐ UNCLASSIFIED/UNLIMITED ☒ SAME AS RPT. ☐ DTIC USERS

21. ABSTRACT SECURITY CLASSIFICATION
Unclassified

22a. NAME OF RESPONSIBLE INDIVIDUAL

22b. TELEPHONE *(Include Area Code)*

22c. OFFICE SYMBOL

DD FORM 1473, 84 MAR — 83 APR edition may be used until exhausted. All other editions are obsolete.

SECURITY CLASSIFICATION OF THIS PAGE
UNCLASSIFIED

PREFACE

We wish to give special thanks to Carol J. Selvey for the acquisition and interpretation of the photoluminescence spectral data.

CONTENTS

FIGURES

I. INTRODUCTION

We have previously suggested[1] that limitations on the purity of undoped GaAs films grown in the Aerospace in-house-built Organometallic Chemical Vapor Deposition (OMCVD) reactor may be due to flaws in the design and construction of the gas handling lines, and to certain instrument imprecision and unreliability. In an attempt to alleviate these problems, plans were formulated for a major revision of the OMCVD reactor system; these included the redesign and replacement of all gas handling lines, the installation of a new mass flow control system, the repositioning of the mass flow control units, and the replacement of the organometallic temperature control baths. We describe here these renovations and upgrades to the OMCVD reactor system, as well as the effect of these modifications on the growth of high-purity GaAs epilayers.

II. SYSTEM MODIFICATIONS

A. GAS HANDLING SYSTEM

Gas lines are known to be a source of potential reagent contamination as a result of the outgassing of impurities from the line walls; thus, it is highly desirable to minimize contact of reagent gases with gas line walls in delivering pure reagents to the reactor chamber. Since the gas handling system of the existing in-house-built OMCVD reactor contained several meters of unnecessary stainless-steel gas line, as well as many unused or dead-end valves as a result of many previous modifications, all existing plumbing was removed and the entire stainless steel gas handling system was redesigned and rebuilt. The new design eliminated all obsolete gas lines and simplified the tubing layout to reduce the overall length of pipe required. In addition, all welded seals were prepared by means of orbital welding, which produces relatively smooth inner wall surfaces at the weld position in order to eliminate potential virtual leaks. Finally, unlike the previous system, which was constructed from a combination of Swagelok, VCR, and pipe fitting connectors, VCR connections were used exclusively in the new system. It is estimated that the remodeling and replacement of the existing gas lines has resulted in a four-fold reduction in gas path length.

B. MASS FLOW CONTROLLERS

As part of the new reactor system's design, a new mass flow control system consisting of a series of high-precision flow control valves and a new control panel was installed. The new control system exhibits a wide variety of parameter options, is extremely easy to operate, and is potentially programmable. This system allows for a more accurate and consistent delivery of gases to the reaction chamber. Another significant alteration to the system was a design change involving the placement of the mass flow control valve units. The new mass flow control valve units have been repositioned from the output side of the organometallic bubblers to the input side of the bubblers. As a result of this change, the bubblers are now operated at a constant pressure of one atmosphere and the organometallic reagents do not

contaminate the mass flow control unit. Prior to this modification, the bubblers were kept at a hydrogen overpressure of 2 to 5 atm, the value of which was determined by a hydrogen pressure regulator at the input side of the bubblers. The imprecision of this regulator allowed the input pressure to vary by 2 to 3 psi during a growth run, thus affecting the concentration of organometallic reagant in the carrier gas flow. In addition, the flow of organometallic reagents through the mass flow control valve units in the previous design caused the capillaries of the units to become clogged occasionally, thus adversely affecting the accuracy of the gas flow and resulting in the need for periodic cleaning.

C. CONTROLLED-TEMPERATURE BATHS

The introduction of accurate and precise quantities of organometallic reagents into the reaction chamber depends on the ability to control the vapor pressure of these reagents within their bubblers precisely and accurately. This requirement demands that the temperature of the bubblers be well controlled. The temperature baths used in the existing system were found to demonstrate temperature fluctuations as great as ±2°C over a 2-hr growth interval, which caused significant variations in the concentration of the organometallic reagent delivered to the reactor during an experiment. On the basis of this observation, the existing temperature baths were replaced with new Neslab baths that control temperatures to ±0.2°C. This feature ensures an accurate, uniform flow of reagent into the reactor during growth.

III. RESULTS

The effect of the reconstruction of the OMCVD system on GaAs epilayer quality was determined by a series of growth experiments carried out after the system was rebuilt. The best material obtained from the old system had been grown with parameter values of V/III = 41, growth temperature = 650°C, and a total gas flow = 3 slpm, which produced an epilayer having a carrier concentration of 2×10^{15} cm^{-3} and a 77 K mobility of ~41,000 cm^2/V-s. The best material grown thus far in the renovated system has been grown under the similar conditions of V/III = 51, growth temperature = 650°C, and total gas flow = 3 slpm. However, the electrical characteristics of the epilayer grown in the redesigned growth system have been found to be vastly superior to those obtained for the earlier sample, with a carrier concentration of 3×10^{14} cm^{-3} and a 77 K mobility of ~68,000 cm^2/V-s. A photoluminescence spectrum of this sample, shown in Fig. 1, confirms the high quality of this epilayer. The luminescence peaks in both the exciton and acceptor regions (shown in detail in Figs. 2 and 3, respectively) are very sharp and well-resolved, which is indicative of high-quality material; this resolution has allowed for the positive identification of the three acceptor impurities in the epilayer as carbon, zinc, and silicon. Out of a series of five additional growth experiments carried out with slight parameter changes, not one sample exhibited a 77 K mobility that was less than 53,000 cm^2/V-s. It is obvious from these results that the reconstruction and upgrading of the OMCVD system has resulted in a cleaner and more precisely controlled environment for the growth of GaAs epilayers.

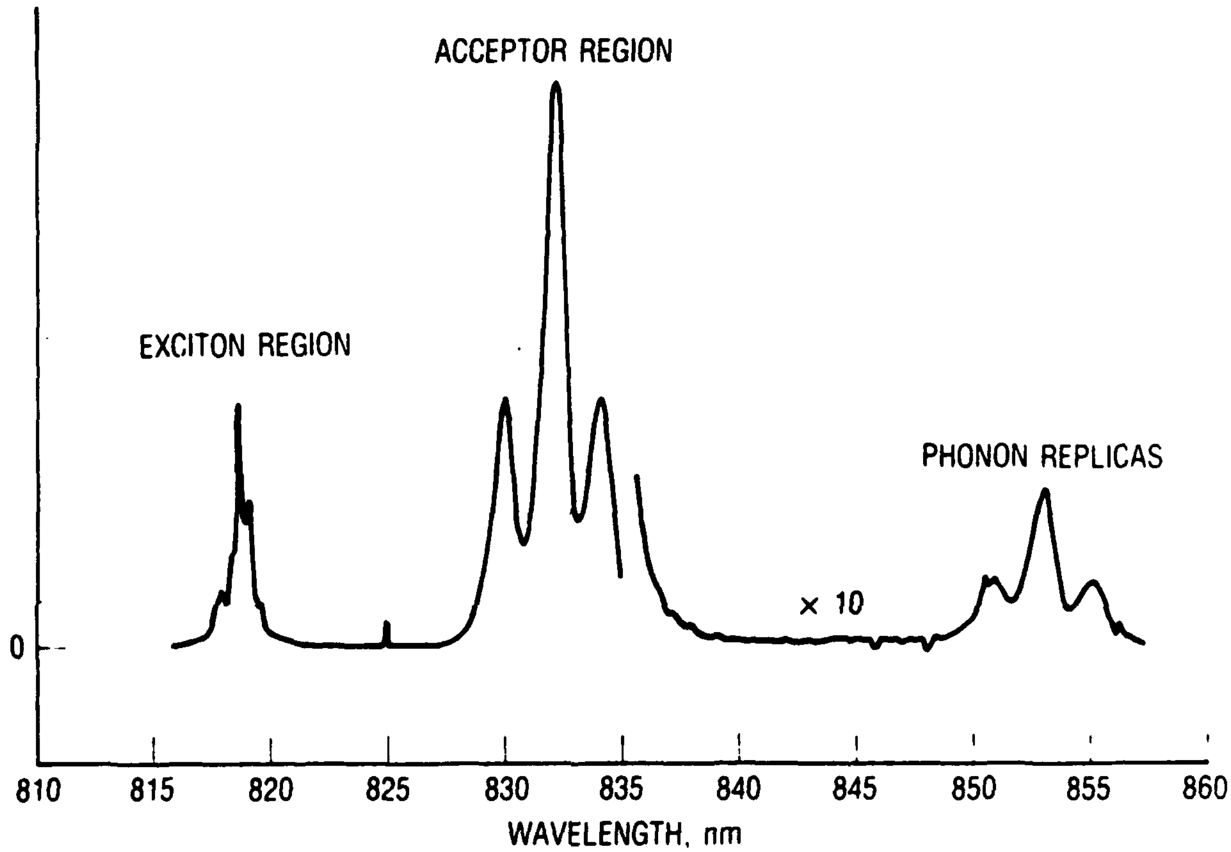

Fig. 1. Photoluminescence Spectrum of OMCVD-Grown Undoped GaAs

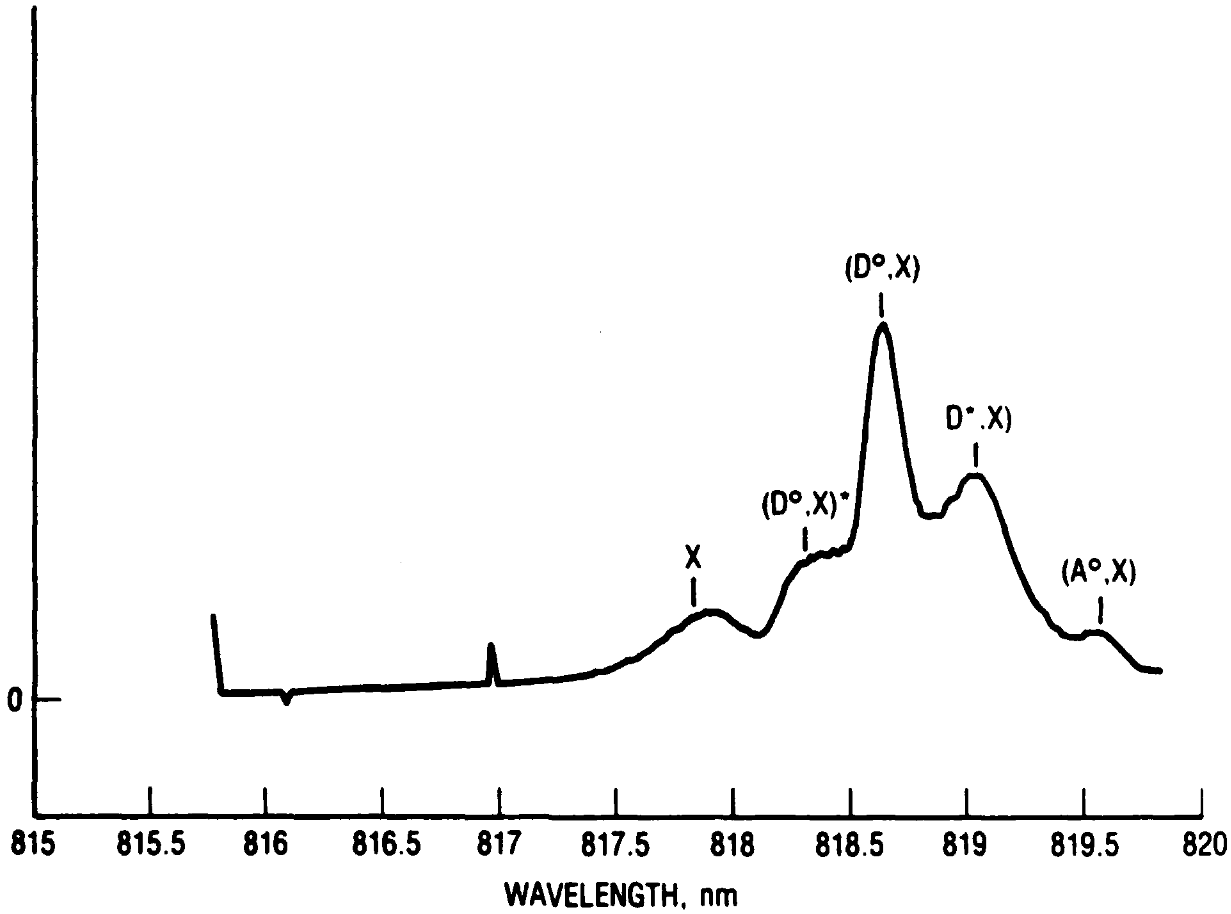

Fig. 2. Exciton Region of GaAs Photoluminescence Spectrum

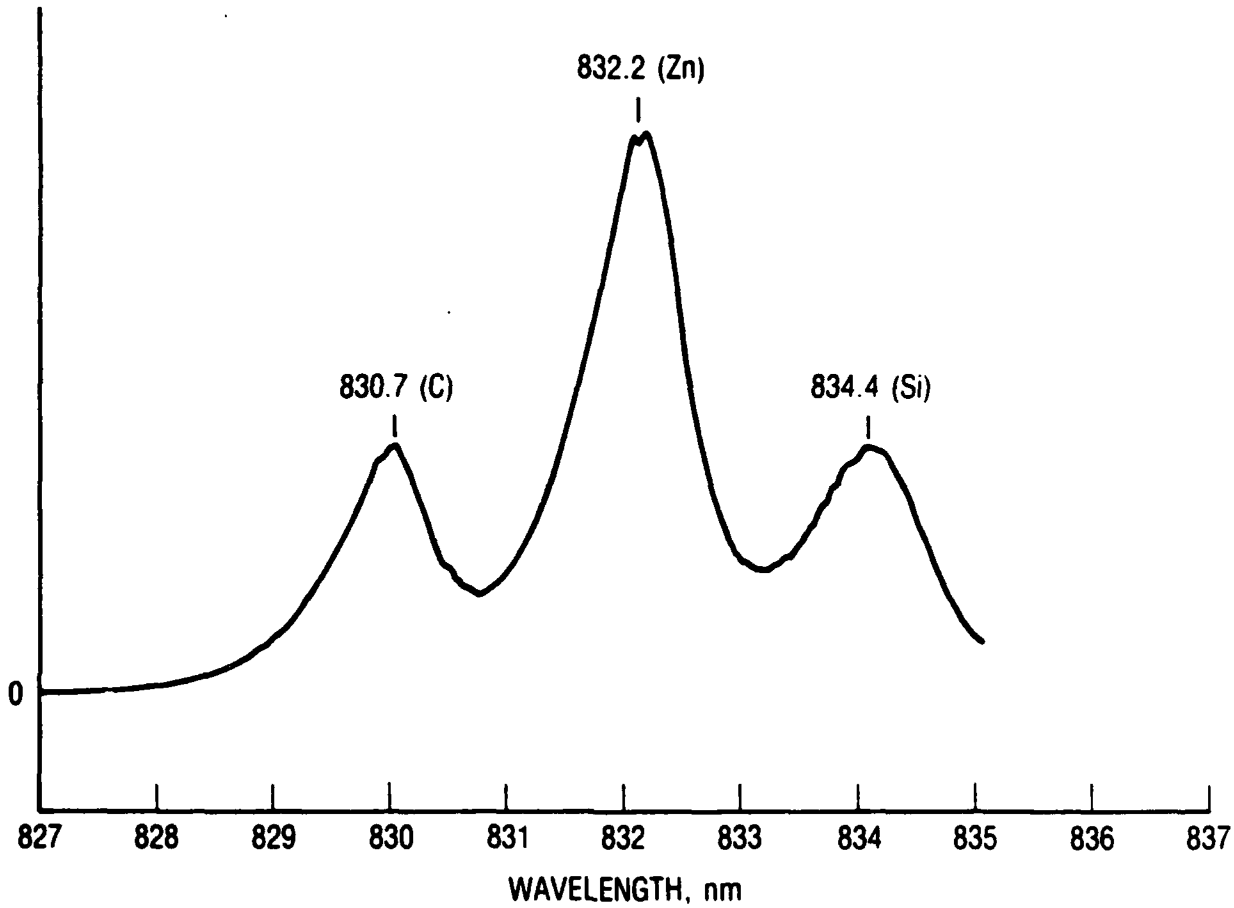

Fig. 3. Acceptor Region of GaAs Photoluminescence Spectrum.

IV. FUTURE PLANS

We plan to continue our efforts in the growth of high-purity GaAs epilayers via the use of new organometallic arsenic and gallium reagents. We have already installed organometallic bubblers containing triethylarsenic and triethylgallium in the renovated system. It is hoped that the use of these reagents will result in a reduction in the amount of carbon found in the OMCVD-grown GaAs epilayers.[2] The use of triethylarsenic in place of arsine will also improve system safety as a result of reduced toxicity.[3] Preliminary growth experiments using these reagents have exhibited promising results.

V. SUMMARY

A complete redesign and reconstruction of the OMCVD reactor gas handling system was carried out, which resulted in a reduction in reagent gas contamination sources and in an improvement in system safety. Both the mass flow control system and the controlled-temperature baths were also replaced with upgraded units that deliver reagents more precisely and accurately to the OMCVD reaction chamber. These changes greatly improved the quality of GaAs epilayers grown in the reactor system, as evidenced by the electrical properties of films prepared prior to and following the renovations. Epilayers grown in the modified reactor system exhibited carrier concentrations that were an order of magnitude lower than those of films grown prior to the system changes, while the mobilities of the newer epilayers were found to be 50 to 70% greater than those of films previously grown. It thus appears that the reactor system changes were essential for the growth of high-purity GaAs epilayers. These changes will allow us to determine whether or not the use of new organometallic reagents can further reduce the incorporation of impurities.

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LABORATORY OPERATIONS

The Aerospace Corporation functions as an "architect-engineer" for national security projects, specializing in advanced military space systems. Providing research support, the corporation's Laboratory Operations conducts experimental and theoretical investigations that focus on the application of scientific and technical advances to such systems. Vital to the success of these investigations is the technical staff's wide-ranging expertise and its ability to stay current with new developments. This expertise is enhanced by a research program aimed at dealing with the many problems associated with rapidly evolving space systems. Contributing their capabilities to the research effort are these individual laboratories:

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